

METHOD #: 413.1	Approved for NPDES (Editorial Revision 1978)
TITLE:	Oil And Grease (Gravimetric, Separatory Funnel Extraction)
ANALYTE:	Oil Grease
INSTRUMENTATION:	N/A
STORET No.	00556

1.0 Scope and Application

- 1.1 This method includes the measurement of fluorocarbon-113 extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the determination of relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter.
- 1.2 The method is not applicable to measurement of light hydrocarbons that volatilize at temperatures below 70°C. Petroleum fuels from gasoline through #2 fuel oils are completely or partially lost in the solvent removal operation.
- 1.3 Some crude oils and heavy fuel oils contain a significant percentage of residue-type materials that are not soluble in fluorocarbon-113. Accordingly, recoveries of these materials will be low.
- 1.4 The method covers the range from 5 to 1000 mg/L of extractable material.

2.0 Summary of Method

- 2.1 The sample is acidified to a low pH (< 2) and serially extracted with fluorocarbon- 113 in a separatory funnel. The solvent is evaporated from the extract and the residue weighed.

3.0 Definitions

- 3.1 The definition of oil and grease is based on the procedure used. The nature of the oil and/or grease, and the presence of extractable non-oily matter will influence the material measured and interpretation of results.

4.0 Sampling and Storage

- 4.1 A representative sample of 1 liter volume should be collected in a glass bottle. If analysis is to be delayed for more than a few hours, the sample is preserved by the addition of 5 mL HCl (6.1) at the time of collection and refrigerated at 4°C.
- 4.2 Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentration over an extended period.

5.0 Apparatus

- 5.1 Separatory funnel, 2000 ml, with Teflon stopcock.
- 5.2 Vacuum pump, or other source of vacuum.
- 5.3 Flask, boiling, 125 mL (Corning No. 4100 or equivalent).
- 5.4 Distilling head, Claisen or equivalent.
- 5.5 Filter paper, Whatman No. 40, 11 cm.

6.0 Reagents

- 6.1 Hydrochloric acid, 1:1. Mix equal volumes of conc. HCl and distilled water.
- 6.2 Fluorocarbon-113, (1,1,2-trichloro-1,2,2-trifluoroethane), b.p. 48°C.
- 6.3 Sodium sulfate, anhydrous crystal.

7.0 Procedure

- 7.1 Mark the sample bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 5 mL hydrochloric acid (6.1) to the sample bottle. After mixing the sample, check the pH by touching pH-sensitive paper to the cap to insure that the pH is 2 or lower. Add more acid if necessary.
- 7.2 Pour the sample into a separatory funnel.
- 7.3 Tare a boiling flask (pre-dried in an oven at 103°C and stored in a desiccator).
- 7.4 Add 30 mL fluorocarbon-113 (6.2) to the sample bottle and rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate, and filter the solvent layer into the flask through a funnel containing solvent moistened filter paper.
NOTE: An emulsion that fails to dissipate can be broken by pouring about 1 g sodium sulfate (6.3) into the filter paper cone and slowly draining the emulsion through the salt. Additional 1 g portions can be added to the cone as required.
- 7.5 Repeat (7.4) twice more, with additional portions of fresh solvent, combining all solvent in the boiling flask.
- 7.6 Rinse the tip of the separatory funnel, the filter paper, and then the funnel with a total of 10-20 mL solvent and collect the rinsings in the flask.
- 7.7 Connect the boiling flask to the distilling head and evaporate the solvent by immersing the lower half of the flask in water at 70°C. Collect the solvent for reuse. A solvent blank should accompany each set of samples.
- 7.8 When the temperature in the distilling head reaches 50°C or the flask appears dry remove the distilling head. Sweep out the flask for 15 seconds with air to remove solvent vapor by inserting a glass tube connected to a vacuum source. Immediately remove the flask from the heat source and wipe the outside to remove excess moisture and fingerprints.
- 7.9 Cool the boiling flask in a desiccator for 30 minutes and weigh.

8.0 Calculation

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$$\text{mg/L total oil and grease} = \frac{R - B}{V}$$

where:

R = residue, gross weight of extraction flask minus the tare weight, in milligrams.

B = blank determination, residue of equivalent volume of extraction solvent, in milligrams.

V = volume of sample, determined by refilling sample bottle to calibration line and correcting for acid addition if necessary, in liters.

9.0 Precision and Accuracy

9.1 The two oil and grease methods in this manual were tested by a single laboratory (EMSL) on sewage. This method determined the oil and grease level in the sewage to be 12.6 mg/L. When 1 liter portions of the sewage were dosed with 14.0 mg of a mixture of #2 fuel oil and Wesson oil, the recovery was 93% with a standard deviation of ± 0.9 mg/L.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 515, Method 502A, (1975).
2. Blum, K. A., and Taras, M. J., "Determination of Emulsifying Oil in Industrial Wastewater", JWPCF Research Suppl. 40, R404 (1968).